

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

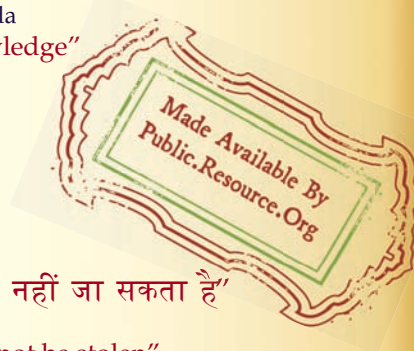
IS 14707 (1999): Methyl Acrylate [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



भारतीय मानक
मिथाईल एक्राईलेट — विशिष्टि

Indian Standard
METHYL ACRYLATE — SPECIFICATION

ICS 71.080.01

© BIS 1999

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Methyl acrylate is produced by the esterification of acrylic acid or by acrylonitrile.

It has versatile use in the manufacturing of various polymeric resins and fibres. It is used for producing polymethyl methacrylate, used as leather binders. It has an important role as water treatment chemicals for purification and it also forms a part of ion-exchange resins.

Methyl acrylate is a flammable liquid. Its vapours can form explosive mixtures with air and are particularly irritant. It should be kept away from heat sources and chemicals that can cause its polymerization violently evolving considerable heat.

Considerable assistance has been derived from the following International specification in preparation of this standard.

ASTM D 1364-1980 Test method for water in volatile solvents

ASTM D 1613-1985 Test method for acidity in volatile solvents

ASTM D 3125-1983 Test method for MEHQ in colourless monomeric acrylate esters and acrylic acids

ASTM D 3362-1984 Test method for purity of acrylate esters by gas chromatography

ASTM D 4709-1987 Specification for methyl acrylate

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHYL ACRYLATE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for methyl acrylate.

2 NORMATIVE REFERENCES

The following Indian Standards are necessary adjuncts to this standard. The standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent revisions of the standards indicated below.

IS No.	Title
265 : 1987	Hydrochloric acid (<i>third revision</i>)
1070 : 1992	Water reagent grade (<i>third revision</i>)
2362 : 1973	Determination of water by Karl Fischer method (<i>first revision</i>)
4905 : 1968	Methods for random sampling
8768 : 1988	Method of measurement of colour in liquid chemical products in hazen units (<i>first revision</i>)

3 REQUIREMENTS

The methyl acrylate shall comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

As agreed to between purchaser and supplier.

4.2 Marking

The material shall be legibly marked with the following information:

- Name of the material;
- Indication of source of manufacture;

- Batch number, in code or otherwise to enable the lot of manufacture to be traced back from records; and
- Net mass of the material.

Table 1 Requirements for Methyl Acrylate
(Clause 3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass, <i>Min</i>	99.5	A	—
ii)	Acidity (as acrylic acid), percent by mass, <i>Max</i>	00.01	B	—
iii)	Colour, Pt-Co scale, <i>Max</i>	20.00	—	8768
iv)	Water, percent by mass, <i>Max</i>	00.20	—	2362
v)	Inhibitors (as methyl etherhydroquinone), ppm <i>Max</i>	120	C	—

4.2.1 BIS Certification Marking

The containers may also be marked with the Standard Mark. The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Sampling shall be done as given in Annex D.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF ASSAY

A-1 GENERAL

This test method covers the determination of the purity of methyl acrylate by gas chromatography and, in addition, provides a means for measuring certain impurities such as alcohols and other esters. Water and acidity are measured by other methods and the results are used to normalize the chromatographic values.

A-2 APPARATUS

A-2.1 Chromatograph

Any gas liquid chromatographic instrument having a flame ionization detector.

Column length	6 m (20 ft)
I.D.	6.4 mm (1/4 in)
Syringe	2 µl
Sample size	1-2 µl
Recorder	A recording potentiometer with a full scale deflection of 1 mV, full scale response time of 2 s or less
Carrier gas	Helium of purity 99.95 mol percent or Nitrogen gas
Column material	Copper/Stainless steel/Aluminium
Liquid phase	Ucon lubricant 50HB 2 000 X or equivalent phase capable of achieving desired separation of various components of interest
Solid support	Chromosorb P-acid washed 45-60 mesh size
Solvent	Methylene chloride, reagent grade

A-2.2 Standards for Calibrations and Identification

Standard samples of all components present are needed for identification by retention time and for calibration of quantitative measurement.

A-2.3 Instrument Conditions

Temperature, °C	110
Column, isothermal	110
Injection port	180

Detector block	240
Carrier gas	Helium
Flowrate ml/min	80
Typical retention time, min	9.6

NOTE — The above gas chromatographic conditions are suggestive. However, other GC with different columns may be used provided standardization/calibrations are done after setting up chromatographic conditions for the required resolution.

A-3 PROCEDURE

Introduce a representative specimen into the chromatograph using sufficient specimen to ensure a minimum 10 percent recorder deflection for a 0.1 percent concentration of purity at the most sensitive setting of instrument. Using the same conditions as for component identification and standardization, record the peaks of all components at attenuation setting that provide maximum peak heights.

A-4 CALCULATION

A-4.1 Measure the area of all peaks and multiply each area by the appropriate detector response factors to correct the difference in response to the components. Calculate the weight percent composition by dividing the individual corrected component areas by the total corrected area. Make corrections to account for water and acidity present.

A-4.2 Calculate the mass percent of each component as follows:

$$\text{Mass, percent} = \frac{(A) \times (100 - C)}{B}$$

where

- A = corrected peak response,
 B = sum of corrected peak responses, and
 C = sum of water and acidity.

A-5 PRECISION AND BIAS

A-5.1 Repeatability

Two results, should be considered suspect if they differ by more than 0.06 percent.

A-5.2 Reproducibility

Two results should be considered suspect if they differ by more than 0.27 percent.

ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF ACIDITY

B-1 GENERAL

This test method covers the determination of total acidity as acrylic acid, in concentrations below 0.05 percent in methyl acrylate. The specimen is mixed with either an equal volume of water or an equal volume of alcohol and titrated with aqueous Sodium Hydroxide solution to the phenolphthalein end point.

B-2 APPARATUS

B-2.1 Burette

10 ml, graduated in 0.05 ml.

B-2.2 Erlenmeyer Flask

250 ml.

B-3 REAGENTS

B-3.1 Alcohols

Refined ethyl or *iso*-propyl.

B-3.2 Phenolphthalein Indicator Solution (10 g/l)

Dissolve 1 g of phenolphthalein in ethyl or *iso*-propyl alcohol and dilute to 100 ml with the alcohol.

NOTE — Bromophenol blue indicator may also be used.

B-3.3 Sodium Hydroxide

Standard solution 0.05 N

NOTE — Alternatively, KOH solution may be used.

B-4 PROCEDURE

Measure into a 250 ml conical flask 50 ml of water, if the sample is water soluble, or 50ml of alcohol, if the sample is not completely water soluble. Add 0.5 ml of phenolphthalein indicator solution. Titrate with water/alcohol with 0.05 N NaOH solution to the first pink colour. Pipette 50 ml of the sample into the flask. Titrate with the 0.05 N NaOH solution to the same first pink colour originally obtained.

B-5 CALCULATION

$$\text{Acidity as acrylic acid, percent by mass} = \frac{VN \times 0.144}{D}$$

where

V = NaOH solution required for titration of the sample,

N = normality of NaOH, and

D = specific gravity of the specimen at the test temperature.

B-6 PRECISION AND BIAS

Repeatability 0.000 8 percent

Reproducibility 0.001 4 percent

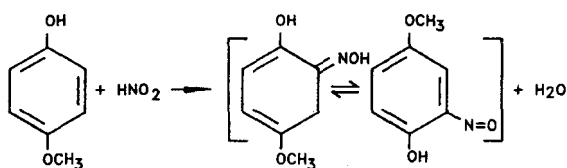
ANNEX C

[Table 1, Sl No. (v)]

DETERMINATION OF INHIBITORS

C-1 GENERAL

This test method covers the determination of monomethylether of hydroquinone (MEHQ) in methyl acrylate. It is applicable to the determination of MEHQ in the concentration range from 0 to 1 200 parts per million. MEHQ reacts with nitrous acid (sodium nitrite in acidic media) to form the nitroso derivative which equilibrates between two structures as given below:



The yellow colour of the nitroso compound is measured spectrophotometrically at a wavelength of 420 nm.

C-2 APPARATUS

C-2.1 Spectrophotometer

With borosilicate glass cells for determining absorbance at 420 nm.

C-2.2 Volumetric Flasks

50 and 100 ml capacity.

C-2.3 Measuring Pipette

5 and 10 ml capacity.

C-3 REAGENTS

C-3.1 Acetic Acid

Glacial.

C-3.2 Monomethylether of Hydroquinone (MEHQ) (4-methoxy phenol)

C-3.3 Sodium Nitrite Solution

Dissolve 2 g of sodium nitrite in water and dilute to 100 ml.

C-4 CALIBRATION

Weigh 0.10 g of MEHQ to the nearest 0.1 mg into a 100 ml volumetric flask containing approximately 50 ml of glacial acetic acid. Mix well until solution is complete then dilute to the mark with glacial

acetic acid. Prepare a series of standards by pipetting 1, 2, 4, 6 and 10 ml portions of the MEHQ solution into respective 50 ml volumetric flasks. Dilute each flask to the mark with glacial acetic acid and mix well. A 10 ml aliquot of each of these standards contains 200, 400, 800, 1 200 and 2 000 µg of MEHQ respectively.

Determine the absorbance each of these standards by pipetting 10 ml aliquots into 50 ml volumetric flasks containing 20 ml of glacial acetic acid. To each flask add 1 ml of 2 percent NaNO₂ solution and dilute to the mark with glacial acetic acid. Mix well and allow to stand for 10 min. With cells appropriate to the instrument, determine the absorbance at 420 nm using acetic acid as the blank.

Construct a calibration curve on rectangular coordinate graph paper by plotting the absorbances of the standards at 420 nm against the micrograms of MEHQ.

C-5 PROCEDURE

Perform analysis in duplicate and carry a blank through the analysis using 49 ml of glacial acetic acid in place of the specimen solution.

Weigh the appropriate amount of specimen (Table 1) into a 50 ml volumetric flask containing 20 ml of glacial acetic acid.

Add 1 ml of 2 percent NaNO₂ solution to the specimen and dilute to the mark with glacial acetic acid. Mix well and allow to stand for 10 min.

Using the procedure followed for the calibration, determine the absorbance of the solution at 420 nm with the blank solution in the reference position. From the calibration curve, determine the micrograms of MEHQ corresponding to the absorbance obtained.

C-6 CALCULATION

Calculate the concentration of MEHQ in ppm as follows:

$$\text{MEHQ, ppm} = \frac{M}{S}$$

where

M = micrograms of MEHQ from calibration curve; and

S = grams of specimen used in the test.

C-7 PRECISION**C-7.1 Repeatability**

Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 4.0 percent relative.

C-7.2 Reproducibility

Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 15 percent relative.

ANNEX D

(Clause 5)

SAMPLING OF METHYL ACRYLATE**D-1 GENERAL**

D-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

D-1.2 Sampling instrument shall be clean and dry.

D-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.4 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed, as thoroughly as possible, by suitable means.

D-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

D-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture and other important particulars of the consignment.

D-1.8 Samples shall be stored in a cool and dry place.

D-2 SCALE OF SAMPLING**D-2.1 Lot**

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

D-2.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately.

D-2.3 The number of container (n) to be selected for drawing the samples shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling

Lot Size	No. of Containers to be Selected
(N)	(n)
(1)	(2)
Up to 25	2
26 to 50	3
51 to 100	5
101 to 300	7
301 and above	10

NOTE — When the size of the lot is three or less, all the containers shall be sampled.

D-2.4 These containers shall be selected at random from the lot and to ensure the randomness of selection, procedures given in IS 4905 may be followed.

D-3 TEST SAMPLE AND REFEREE SAMPLE

D-3.1 From each of the container selected as in **D-2.3**, draw with an appropriate sampling instrument small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

D-3.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

D-3.3 From the samples (*see D-3.2*) representing different containers selected in **D-2.3**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient to carry out testing for the characteristics specified. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

D-3.4 The referee samples consisting of a composite sample shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to

between the two. This shall be used in case of any dispute between the two.

D-4 TESTS

D-4.1 Tests for all the characteristics shall be conducted on the composite sample.

D-5 CRITERIA FOR CONFORMITY

D-5.1 For declaring the conformity of a lot to the requirements of all other characteristics (*see D-4.1*) tested on the composite sample, the test results for the characteristics shall satisfy the relevant requirements given in Table 1.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 9 (1451).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :

Telephone

Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	{ 323 76 17 323 38 41
Eastern	: 1/14 C. I. T. Scheme VII M, V. I. P. Road, Kankurgachi CALCUTTA 700 054	{ 337 84 99, 337 85 61 337 86 26, 337 91 20
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160 022	{ 60 38 43 60 20 25
Southern	: C. I. T. Campus, IV Cross Road, CHENNAI 600 113	{ 235 02 16, 235 04 42 235 15 19, 235 23 15
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400 093	{ 832 92 95, 832 78 58 832 78 91, 832 78 92
Branches	: AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM.	